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## TECHNICAL REPORT

CONTROL OF STRESS CORROSION-  
2ND INTERIM REPORT

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By

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R. H. Wolff

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Department of the Army Project No. 1CO-24401-A110

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Rock Island, Illinois

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## ABSTRACT

This study of the control of stress corrosion cracking susceptibility of steels by application of protective coatings was designed to use abrasive blasted specimens to simulate more nearly the surfaces and conditions of a manufacturing operation. Bent beam specimens of 4130, 6150, and 18% nickel maraging steel were prepared at yield strength levels of 204, 231, and 316 ksi respectively for test at 75% of yield strength. Test atmospheres were outdoor, high humidity and salt spray (5%), and cycles of these alternating between salt spray, humidity and air. Cycle tests produced more rapid failure than single environments. Abrasive blasting extended the time to failure as compared to non-blasted uncoated specimens. Coated specimens were electroplated with zinc, zinc phosphatized, or brushed with zinc filled paint. Specimens of 4130 steel had not failed in over a year in outdoor exposure and 6 months in high humidity. Failures were noted with all the materials in cycle tests with indication of extended time to failure as a result of using zinc filled paint.

## RECOMMENDATIONS

It is recommended that high strength materials be abrasive blasted prior to final finish application to make use of the improvement in stress corrosion cracking resistance noted in this work.

# CONTROL OF STRESS CORROSION - 2ND INTERIM REPORT

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## CONTROL OF STRESS CORROSION - 2ND INTERIM REPORT

### OBJECT

To study the reduction of susceptibility to stress corrosion cracking of high strength steels by use of protective coatings.

### INTRODUCTION

This report is a continuation of a study<sup>(1)</sup> of the control of stress corrosion cracking susceptibility of steels by application of protective coatings. Work was designed to use abrasive blasted specimens to simulate more nearly the surfaces and conditions of a manufacturing operation. Initial work was conducted using specimens of aircraft quality 4130 alloy sheet steel, heat treated to approximately 200 ksi yield strength. Zinc was chosen as the basis of protective coating and was applied to abrasive blasted bent beam specimens as follows: by zinc electroplating, zinc phosphatizing, and by zinc dust dispersed in a vehicle. This work reports the results of tests.

### EXPERIMENTAL PROCEDURE

Strips of 4130, 6150 and 18% nickel maraging steel were prepared as bent beam specimens .065 x 1 x 9 inches. Yield strength determinations were made (see Table I) and by the method of Phelps et al<sup>(2)</sup> specimen lengths determined and cut to produce a tensile load of 75% of yield strength in the bent beam fixture. Specimens were uniformly steel grit blasted and coated except as tested bare for control. Tests were run in duplicate or triplicate as exposure space allowed.

Protective coatings used were from a hot zinc phosphatizing production bath, a conventional zinc cyanide electroplating solution, and a proprietary paint of zinc dust dispersed in a vehicle.

Test environments used were an outdoor semi-industrial exposure area approximately 300 feet from the Mississippi River; high humidity (100% RH at 100°F.); and 5% salt spray fog (Method 811.1 of Federal Test Method Standard 151a).

After the initial series of tests, additional tests were begun using cycles in which specimens were repeatedly alternated between salt spray, humidity cabinet and standing in air. These tests provided cycles such as 52 hours in salt spray, 52 hours in high humidity and 64 hours (over

TABLE I

TENSILE TEST OF BLASTED AND UNBLASTED SPECIMENS

Steel	Yield Strength ksi	Ultimate Strength ksi	Modulus of Elasticity x10 <sup>6</sup>
<u>4130</u>			
1	205	251	28.9
2	206	252.5	30.0
3B	204	252.5	30.2
4B	203	252	29.7
5	206	252.5	-
6	201	252	29.6
Average	204	252	29.7
<u>6150</u>			
1B	228.7	244.3	
2	232.5	244.6	
3B	231.8	245.3	
4B	228.3	241.2	
5	236.1	247.7	
6	231.6	245.8	
Average	231.5	244.8	30.

Specimens marked "B" were steel grit blasted.

18% Ni

1	301.7	304	
2	324.3	326.8	
3B	312.7	316.	
4B	324.	325.6	
Average	315.8	318.6	27.6

Mill Certified Analysis of 18% Nickel Maraging Steel

C	Mn	P	S	Si	Cr	Ni	Al
.027	.030	.007	.006	.040	.015	18.95	.13
Mo	Zr	Ti	N <sub>2</sub>	Co	B		
5.05	.009	.81	.0044	8.75	.0023		



week end) standing in air. The order of salt spray or humidity was varied for different tests. Other cycles were exposure to salt spray during working hours alternated with standing in air for nonworking hours. This cycle was 8 hours in salt spray, 16 hours in air for 5 days, and standing in air over weekends. In such tests, the salt solution was allowed to dry on the specimens without rinsing.

## RESULTS AND DISCUSSION

Specimens referred to as "bare" were grit blasted and uncoated. Those called "as heat treated" were not given any further processing after the heat treatment to provide the strength level. These specimens were not scaled or otherwise unsatisfactory in appearance to the eye. Heat treatment was accomplished in a neutral, controlled atmosphere furnace with carbon potential similar to the carbon content of the material being treated.

Except for "as heat treated" specimens, which failed in all environments, there was no significant evidence to indicate stress corrosion cracking susceptibility of 4130 steel in the outdoor or humidity cabinet exposures. Specimens in test from 6 months to over a year have not failed. In contrast, the exposures which included salt spray, either as single exposure or as part of a cycle, produced many failures. As shown in Table II, failure times varied among test procedures. The general trend suggested that cycle tests produced failures in less time than single exposure tests.

Behavior of phosphatized specimens as compared with bare specimens gave no easily defined difference in susceptibility. Phosphatized specimen failure times were generally longer, but in one test were shorter than for bare specimens. This does not show any protective characteristic of the phosphate coating with regard to suppression of stress corrosion cracking. At the same time the evidence does not indicate a trend of increased susceptibility as a result of phosphatizing. This latter possibility has been suggested in the literature.<sup>(3)</sup> The improvement of performance with supplementary coatings over phosphatized steel is an accepted fact. The ability of such coatings to provide a paint base without inducing an increase in stress corrosion cracking may be useful in further work.

Zinc plated specimens suffered from hydrogen embrittlement, and a number were broken within hours or a few days after mounting for test. Those which survived the first few days resisted failure.

TABLE II

## EXPOSURE RESULTS OF 4130 BENT BEAM SPECIMENS

	Outdoor		Humidity Cabinet		Salt Spray	
As heat treated	3/3*	7 wks	6/6	7 days	1/2	13 days
Blasted-uncoated	1/6	NF** 54 wks	0/3	NF 25 wks	2/3	14 wks
Zinc phosphatized	1/6	" "	0/3	" "	2/3	15 wks
Zinc plated	0/5	" "	0/3	" "	1/2	3 days
Zinc dust paint	0/6	" "	0/3	" "	0/3	NF 25 wks
Cycle	104 hrs HC		52 hrs SS		Work days SS	
Test	64 hrs SS		52 hrs HC		Nights and weekends	
			64 hrs Air		in air	
Blasted-uncoated	2/3	27 days	3/3	11 wks	2/2	20 days
Zinc phosphatized	1/3	15 wks	3/3	9 wks	2/2	33 days
Zinc plated	2/3	5 hrs	-	-	-	-
Zinc dust paint	0/3	NF 18 wks	-	-	1/2	7 wks

\* 3 breakages out of 3 specimens in group.

\*\* No failure attributed to stress corrosion.

The zinc dust dispersed paint specimens also resisted failure very well.

The specimens of 6150 and 18% nickel maraging steel, at higher strength levels than the 4130, showed failures in all areas tested. (See Table III) As these materials were received late in the year, only cycle tests were conducted.

The simple use of abrasive grit blasting was an effective pretreatment. This was shown by the greatly increased time to failure between as heat treated and bare specimens for both materials.

The lengthening of failure time is not the final objective of stress corrosion work. Any failure that could be predicted in the expected life of a structure should be considered intolerable. However, failure times do propose directions in which favorable study can be undertaken.

Failure times of phosphatized and zinc dust dispersion coated specimens were longer as compared to the performance of the bare specimens. This suggests again that use of these processes and coatings did not accelerate failure in a susceptible material. It has been noted in the literature that hydrogen liberated during the sacrificial corrosion of such materials as zinc may contribute to brittle fracture failures.

The delay in the failure occurrence as result of abrasive blasting should not necessarily be interpreted as a change in susceptibility to stress corrosion cracking. It is probable that the treatment resulted in a surface barrier effect. For a susceptible material, coatings and surface treatments may act either to intensify or accelerate cracking, or to suppress or retard the cracking process by physical or chemical means. The result noted here indicates the modifying effect of surface treatment should be given further study.

### CONCLUSIONS

The alternate use of exposure conditions in cycles as opposed to a single test exposure condition produced more rapid failures. This is desirable for accelerated test purposes.

Specimens of 4130 steel at 204 ksi yield strength, either coated or bare, were not susceptible to stress corrosion cracking when tested at 75% of yield strength in high humidity or in outdoor exposure. They did fail in

TABLE III

EXPOSURE RESULTS OF 6150 AND MARAGING STEEL BENT BEAM SPECIMENS

	52 hrs HC	52 hrs SS	64 hrs Air	Work Days SS Nights and Weekends in Air	Work Days in Air Nights and Weekends in SS
<u>6150</u>					
As heat treated	3/3*	3 days		-	-
Blasted-uncoated	3/3	2 wks		2/2 19-36 days	-
Zinc phosphatized	3/3	6 wks		2/2 21-49 days	-
Zinc dust paint	2/3	8 wks		-	2/2 6 wks
18% nickel maraging steel					
As heat treated	3/3	40 hrs		-	-
Blasted-uncoated	3/3	8 days		2/2 30 days	-
Zinc phosphatized	-	-		2/2 1-8 wks	-
Zinc dust paint	-	-		-	1/2 17 days

\* 1st figure is number of failures 2nd figure is test specimens in group.

salt spray, or in cycle tests which included salt spray.

Specimens of 6150 at 231 ksi and 18% nickel maraging steel at 316 ksi failed in cycle tests of salt spray. The use of phosphatized coats and zinc dust dispersed paint retarded the failure times as compared to uncoated specimens.

Abrasive blasting of specimens extended the time to failure over "as heat treated" specimens.

Use of zinc phosphatizing did not appear to induce greater susceptibility to failure as compared with uncoated specimens.

### LITERATURE REFERENCES

1. "Control of Stress Corrosion - Interim Report,"  
R. H. Wolff, Rock Island Arsenal Report No. 63-3890,  
26 Nov. 63.
2. "Stress Corrosion of Steels for Aircraft and Missiles,"  
E. H. Phelps and A. W. Loginow, Corrosion, Vol. 16,  
No. 7 (1960).
3. "Stress Corrosion Cracking of High Strength Steels  
and Alloys--Artificial Environments," Mellon Institute  
Contract No. DA 36-034-ORD-3277RD (Frankford Arsenal).

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